PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appln. No.	:	10/803,306	)
Applicant	:	Glenn C. Calhoun et al	)
			)
Filed	:	March 18, 2004	)
Title	:	Process for Preparing an Aqueous Dispersion of a Quaternary Ammonium Salt Containing Vinyl Copolymer	)
			)
TC/A.U.	:	1713	)
Examiner	:	Harlan, Robert D.	)
			)
Docket No.	:	100-00268	)

CERTIFICATE OF ELECTRONIC SUBMISSION

I hereby certify that this correspondence is being submitted electronically with the United States Patent and Trademark Office's electronic filing system (EFS Web) on this 1st day of April, 2009.

/Marie Mikolainis/ 4-1-09

Marie Mikolainis Date

SECOND DECLARATION OF GLENN C. CALHOUN

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Glenn C. Calhoun , hereby declare that:

1. I am one of the joint inventors of the subject matter defined by originally filed claims 1-24 and described in the specification of the above-identified patent application.

2. I, together with Rodney M. Weston, the other joint inventor, conceived and reduced practice the invention defined by claims 1-24 of the present patent application.

3. I received a Ph.D. in organic chemistry from the University of Illinois in 1986.

4. I was employed at S.C. Johnson & Son, Inc. from 1986 to 1999 in the field of polymer chemistry, and I joined Bostik, Inc., the assignee of record of the above-identified patent application, in 1999 to work in the field of adhesive polymer chemistry, where I am currently employed.

5. By virtue of my education and practical experience, I believe I am thoroughly familiar with the subject of polymerizing polymers, especially the process set forth in original claims 1-24 filed in the above-identified patent application.

6. In response to the most recent rejection of our application in the Patent Office Action dated October 2, 2008, I performed a set of experiments to compare our process set forth in claim 1 of the present '306 patent application with the process set forth and described in Chang et al U.S. Published Patent Application No. 2003/0032352A1 to demonstrate that steps E and F in claim 1 are critical to our claimed process and not merely optimization steps having no effect on the claimed process as alleged by the U.S. Patent Examiner.

Our Claimed Process

7. Steps A-D: Acetone (288.21 grams) and water (96.07 grams) were mixed in a flask equipped with a thermocouple, a stirrer having multiple paddles, and a condenser. The acetone and water solution was cooled in an ice water bath and deoxygenated by bubbling dry nitrogen through the solution for 20 minutes. After this time, the solution was heated to its boiling point, ca. 140°F under a nitrogen atmosphere. A first monomer mixture was prepared by mixing Adamquat MC-80 (39.42 grams), a monomer containing quaternary ammonium salt functionality, and deionized water (6.35 grams). A second monomer mixture was prepared by mixing methyl acrylate (335.99 grams), acetone (42.68 grams) and Vazo 52 (3.29 grams), a free radical initiator. The two monomer mixtures were added to the acetone/water solution, with continuous mixing by the stirrer, over a three hour period, maintaining the temperature at the 140°F boiling point, and a polymer solution was formed. After the addition of the two monomer mixtures was complete, the mixture was maintained at the 140°F boiling point with continuous stirring for an additional 2 hours to complete the polymerization. The resultant mixture was about 820 grams of polymer solution containing 45% quaternary ammonium salt containing vinyl copolymer and 55% of a solvent mixture of acetone and

water (75% acetone, 25% water). The viscosity of the polymer solution was about 8000 centipoise.

8. Steps E-G: The 45% polymer solution was diluted with 1100 grams of deionized water and the polymer was completely dispersed by mixing with the stirrer. As a result of adding the water, the total polymer solids content was reduced to about 19% and the solvent composition was now about 22% acetone and 78% water. The viscosity of the mixture was less than 100 centipoise. The dispersion was gradually heated to about 96°C over a 4 hour period and the distillate was collected using the condenser. The final product was a 23% polymer solids dispersion. The remaining 77 percent contains about 97.5% water and 2.5% acetone. The final viscosity of the polymer dispersion was ca.150 centipoise.

9. As described in examples 2-5 of our application, the quantity of collected distillate is high enough and the acetone percentage is high enough for this material to be reused in subsequent batches without significant additions. We have used this process on a commercial scale for about 1.5 years. The distillate composition is typically around 80% acetone and only requires a small amount of water to reduce the content back to 75% for the next batch.

Chang et al '352 Application Comparison

10. Steps A-D were repeated to produce the same quaternary ammonium salt containing vinyl polymer solution. However, step E of claim 1 (the addition of water) was not performed.

11. The temperature of the polymer solution was raised gradually to remove the solvent. The material was heated with a water bath set at 96°C with continuous mixing by the stirrer. A water bath was used as an external heat source, because the expected thick mixture would damage or break an internal thermocouple or thermometer. At temperatures greater than 100°C, we have observed decomposition of the quaternary ammonium salt, so the temperatures were maintained below this level. Over a 2 hour period, the product became progressively thicker and more difficult to mix. Toward the

end of the two hours, the product became so viscous that it pulled away from the sides of the flask and formed a ball around the stirring shaft and paddles. At two hours, the product became so thick that the stirring motor could no longer maintain the same speed. The process was stopped to avoid damage to the stirring motor. The product was essentially a semi-solid mass wrapped around the stirrer shaft and paddles. The flask was broken to analyze the resulting polymer mixture. The solid content of the final polymer mixture was found to be 79%. The material still contained 21% solvent.

12. A large quantity of additional solvent would be needed to reuse the distillate collected from the process at this point. Reclaiming the remainder of the solvent from this very viscous semi-solid material would require much longer times or higher temperatures, which would degrade the polymer. Dispersing the neat polymer in water also would require a large amount of time because of the high molecular weight, the almost solid nature of the polymer, and the low surface area of the large, single polymer chunk.

13. Conclusion: The addition of a large amount of water in step E of claim 1 of our process is the critical step for maintaining a low viscosity needed for a large scale commercial batch process to produce thousands of gallons of product. The unexpected result was the achievement of two seemingly opposing process goals: the ability to easily remove substantially all of the acetone and the ability to recapture acetone rich distillate that can be reused in subsequent batches. By adding the water before the distillation process, the polymer always remains in a low viscosity dispersion, which makes the distillation easier to control and keeps the distillation temperature low to prevent degradation of the polymer. Thus, the desired aqueous dispersion is produced without having to re-disperse the neat polymer.

In contrast, the process described in Chang et al is not able to achieve either of these results. More than 20% of the original solvent remains in the polymer under laboratory conditions. This deficit would also require a significant amount of additional solvent to reuse the distillate that is collected. In a large scale commercial process, these

deficiencies would become greater. Most commercial scale equipment would not be able to handle the very high viscosity of the product in Chang et al's process.

14. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title XVIII of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issued thereon.

April 1, 2009
Date

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